

PROJECT TITLE : ANALYTICAL INVESTIGATIONS
PERIOD COVERED : JANUARY 7th - 25th, 1980
WRITTEN BY : E. LECOULTRE

GC/MS RESEARCH

Eastman's Triacetin

The impurities (or additives?) in ESTROBOND B (1) have been identified by GC/MS and structures assigned by fragmentation.

Peak with RT 13.01 min: Glycerol-2-propionate-1,3-diacetate, $C_{10}H_{16}O_6$ (232). MS: 159 ($M - CH_3COOCH_2$, 24), 57 ($CH_3CH_2CO^+$, 100), 43 (CH_3CO^+ , 31).

Peak with RT 13.26 min: Glycerol-1-propionate-2,3-diacetate, $C_{10}H_{16}O_6$ (232). MS: 159 ($M - CH_3COOCH_2$, 20), 145 ($M - CH_3CH_2COOCH_2$, 14), 57 ($CH_3CH_2CO^+$, 78), 43 (CH_3CO^+ , 100).

Concentration of the isomers in ESTROBOND B are 0.2 and 0.4 %, resp.

Peak with RT 4.45 min reported to be ethylacetate (2) was now identified as 1,3,5-trimethyl benzene (mol.wt.120). MS 121 ($M^+ + 1$, 5), 120 (M^+ , 59), 105 ($M - CH_3$, 100), 91 ($C_7H_7^+$, 10). 1,3,5-trimethyl benzene is not an impurity in ESTROBOND B but of the solvent acetone used for the GS samples.

7

The formation of both isomers in the esterification reaction of crude diacetin with propionic acid (H_3PO_4 as catalyst) could be confirmed by GC/MS.

The large scale synthesis of the isomer mixture is in progress.

E. Lecoultre

REFERENCES

- (1) E. Lecoultre, PME Research Lab. Monthly Progress Report, December 1979, p. 12, Fig. 1.
- (2) Y. Genoud, PME Research Lab. Monthly Progress Report, December 1979.